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METHOD OF CALIBRATION OF IONIZATION VACUUM GAUGES

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METHOD OF CALIBRATION OF IONIZATION VACUUM GAUGES

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Indian Standard

METHOD OF CALIBRATION OF IONIZATION VACUUM GAUGES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 November 1976, after the draft finalized by the Chemical Engineering Sectional Committee had been approved by the Mechanical Engineering Division Council.

0.2 The general method of calibration of a vacuum gauge, which is compatible with the pressure range and the nature of the gases considered, is covered in IS : 8244-1976*. The purpose of this standard is to supplement IS : 8244-1976* in the case where the vacuum gauges to be calibrated are ionization gauges. The reference gauge is either an absolute gauge or a gauge which has been calibrated itself and adapted to this purpose, and is of approved principle and type.

0.3 When the vacuum gauges to be calibrated are assumed to be linear in the pressure range to be explored, it may be much more economical to check the linearity and only to carry out the calibration proper for a pressure value within the linear range, which gives the constant (sensitivity) of the gauge.

0.4 This standard deals only with the establishment of a calibration law for gauges in given conditions. For certain types of gauges, it may be necessary to measure certain other characteristics, such as, for example, the modulation factor for modulation Bayard-Alpert gauges; these supplementary measurements will be dealt with in separate standards.

0.4.1 Furthermore, measurements for type definition may involve noting different characteristics of the gauge the measurement of which is not necessary for the calibration; the conditions in which these values shall be recorded will be given in special standards.

0.5 International system (SI) units of pressure have been used in this standard. The relation of this unit to other units is given below:

1 pascal (Pa) = 1 newton/square metre (N/m^2)
1 torr = 1 mm of Hg = 133·322 Pa

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*Method of calibration of vacuum gauges by direct comparison with reference gauges.

0.6 This standard is based on Draft International Standard ISO/DIS 3568 ' Ionization vacuum gauges—calibration by direct comparison with a reference gauge ' issued by the International Organization for Standardization.

0.7 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard specifies a method for calibrating an ionization vacuum gauge by direct comparison with a reference gauge.

The method is applicable to ionization vacuum gauges of the following recognized types:

a) Various models of hot-cathode vacuum gauges,
b) Triode-type gauges,
c) Bayard-Alpert gauges (including the fine collector gauge),
d) Modulation Bayard-Alpert gauges,
e) Hidden collector or extraction Bayard-Alpert gauges,
f) Ionic beam deviating gauges,
g) Hot-cathode magnetrons,
h) Orbitron-type gauges,
j) Cold-cathode gauges which may include very different structures,
k) Gauges with ionizing particle radioactive sources, and
m) So-called ' high-pressure ' hot-cathode ionization gauges.

1.1.1 Each type of gauge generally corresponds to a field of application to be specified by the manufacturer and reference shall be made to the latter's instructions.

1.1.2 The method is not limited to a particular pressure range but its application limits are fixed by those of the gauges used, the measuring equipment being compatible.

2. PRINCIPLE

2.1 The gauge or gauges to be calibrated and the reference gauge are connected as given in Appendix A to a test chamber in which the required pressures are produced.

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*Rules for rounding off numerical values (*revised*).

2.2 Satisfactory operation assumes the achievement of a uniform and stable distribution of gas in the measuring volume at the pressure tappings on this volume, that the pressures are close enough to enable comparisons to be made with the required accuracy. This presupposes the observance of certain conditions which are examined in **3**.

3. APPARATUS

3.1 Reference Gauges — one or more for use in the range of pressures within which the vacuum gauge is to be calibrated.

3.2 Vacuum System — meeting the requirements given in **3.2.1** to **3.2.6.1**.

3.2.1 The test chamber shall have a volume at least 20 times the total volume of the gauges and any associated plumbing connected to it.

3.2.2 The test chamber shall have a shape such that the ratio of wall surface to volume is the minimum practical (in glass apparatus, for example, a sphere) but not greater than that of a right cylinder having a length equal to four times its diameter.

3.2.3 Communication between the test chamber and the rest of the vacuum system shall be effected in such a way that the incoming stream does not impinge on any of the test or reference gauges or openings thereto.

3.2.4 The reference gauges and gauges to be calibrated shall be connected to the test chamber in an arrangement in which pressure and temperature differentials do not introduce appreciable errors. The geometry of the tubing connecting the measuring chamber with the gauges shall be such that a molecule of gas emanating from the measuring chamber with the gauges shall be such that a molecule of gas emanating from the measuring chamber cannot penetrate the active zone of a gauge without having encountered a wall at least once.

3.2.5 The various gauges may show phenomena of sorption or desorption. In order to minimize the unbalanced pressure distribution resulting from these effects the conductance of the tubing connecting the measuring chamber and the gauges shall be about 100 times the volume rate of flow corresponding to the combined effect of these sorptions and desorptions this usually results in a conductance of at least several litres per second.

3.2.6 The base pressure (that is, when no gas is introduced) shall not exceed two percent of the lower limit of the calibration range as indicated either by the reference gauge or by the gauge to be calibrated. Care shall be taken to ensure that the simultaneous functioning of the reference gauge and the gauge to be calibrated does not result in any discernible mutual influence on their respective readings in steady operation.

3.2.6.1 If simultaneous calibrations are envisaged, it will be necessary to check by means of cross-controls that, in steady operation, there is no appreciable interaction. If such interaction exists, additional precautions shall be taken.

3.3 Ionization Gauge — acting as a monitor for the chamber pressure.

3.4 Buffer volume and monitor gauge for pressure indication in this buffer volume (*see* Appendix A). This material is optional.

3.5 Calibration Gas

3.5.1 The quality of the gas shall correspond to a maximum content of impurities of 0·1 percent by volume.

3.5.2 The device for introducing the gas into the apparatus from the supply container shall be constructed and used in such a way that the purity rate is maintained in practice. Where practicable, the purity of the gas in the system shall be monitored by a partial pressure gauge.

3.6 Thermometers

3.6.1 The ambient temperature around the measuring chamber and the gauges shall be determined by means of thermometers suitably positioned and protected from radiation with an accuracy better than 1°C.

3.6.2 The temperature of the measuring chamber shall be measured by means of thermometers in thermal contact with it with an accuracy better than 1°C.

3.6.3 Both the reference gauges and the gauges to be calibrated shall be placed in still air and, if necessary, protected in such a way that their temperature in stationary conditions, conditioned by their operation and by the ambient temperature, remains constant within the limits specified by their respective manufacturers.

4. PROCEDURE

4.1 In a general way the assembly and preparation of the measuring apparatus and the mounting of the gauges, either reference gauges or gauges to be calibrated, shall be effected according to accepted practice and in agreement with the manufacturers' prescriptions.

4.2 After assembly, pumps are started in the planned order. After a reasonable pumping time, leakage if any, shall be located and eliminated before proceeding with the operations.

4.3 In general the basic pressure specified in **3.2.6** cannot be reached without stoving. For checking pressure in the system, gauge M_C shall be usable during stoving. It shall be ascertained that during heating the pressure does not exceed the maximum allowed by the manufacturers of the various elements of the equipment.

4.4 It is not possible to describe a general procedure for de-gassing the installation and measuring tubes. This shall be established for each installation, bearing in mind that the aim is to obtain a state of cleanness and lack of contamination as satisfactory as possible.

4.5 Gauges shall be de-gassed according to the manufacturers' recommendations, when the chamber is still hot (at about 150°C) and shall be reheated, to about 250°C if required at the end of the de-gassing operation. De-gassing shall not exceed the upper pressure limit (between 10^{-3} and 10^{-4} Pa) to avoid damaging the gauges (ionic bombardment of cathodes, metal spraying, colouring of electrodes) and polluting the walls of the assembly.

Then the isolating resistance of the electrodes and particularly that of the ion collectors shall be checked which shall not be less than a specified value given by the manufacturer.

4.6 The operation of gauges that are to be used simultaneously shall be maintained at the maximum value of the electronic current.

4.7 The measuring gas is introduced and its flow is controlled to give a pressure reading close to the lowest measuring pressure.

4.8 The feed voltages and electronic current shall be adjusted to the required value; if calibrations are carried out with several electronic currents, a start shall be made with the greatest electronic current.

4.8.1 In the case of a complete gauge assembly, the alternative voltage input shall be adjusted to its nominal value.

4.8.2 In the case of a gauge tube only*, the various potentials applied to the electrodes shall be adjusted to the required values.

4.8.3 In both cases, the electronic current is adjusted to the required value. The readings on the measuring instruments shall be adjusted, stabilized so as to obtain a constant ionic current to the nearest one percent for an adequate period (about 5 min).

4.9 The zero setting of the amplifier is checked and adjusted, if required. The indications provided by the measuring instrument of the gauge to be calibrated and that of the reference gauge are read. The values are noted and the ambient temperature is also noted.

4.10 The operations of **4.8** and **4.9** are repeated twice for each calibration point (three measurements in all).

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*Keeping the isolation of the gauge collector within the specified limits, it is ensured that the impedance of the inlet circuit of the measuring ion current amplifier enables a correct measurement of this current to be made.

4.11 The pressure in the system is increased so as to have a sequence of at least three calibration points for each range of the instrument (or three calibration points for each power of ten if the range covers more than one decade). These points should correspond to regularly spaced values of the pressure for the same adjustment of the variable parameters of the gauge. More points may be necessary in the regions where the calibration law does not follow proportionality.

In the case of a logarithmic-scale gauge, one point per range is sufficient.

4.12 If a calibration at other electronic current values is required, all the operations are repeated. First of all, at the lowest pressure, the proportionality between the ionic current and the electronic current at constant pressure is checked, taking account of the first calibration. If necessary, de-gassing is carried out.

4.13 It is advisable to check the heating power of the cathode, if any.

4.14 It is desirable that the quantities from which pressure values are derived should be recorded in an almost continuous way and that the selected values involved in the definition of the calibration points correspond to periods of good stability.

5. PRESENTATION OF RESULTS

5.1 The following particulars shall be stated:

a) Identification of the gauges or gauge tubes including the reference gauge, by the manufacturer's name, type of gauge, series and serial number in the series for the type concerned;
b) The date of calibration;
c) The nature of the gas employed;
d) The methods used for checking the requirements of **3.2.6**, and the anomalies found;
e) The temperature of the measuring chamber and the ambient temperature;
f) The basic pressure;
g) An outline diagram of the calibration bench with reference to the position and orientation of the gauge being calibrated. In the case of a naked structure gauge, a complete drawing of the envelope in which it has been placed for connection to the bench (*see* Appendix A); and
h) The dimensions and nature of the connection of the gauge to the apparatus.

NOTE — From the readings, the true pressures corresponding to each measuring point are calculated.

5.2 The characteristics given in **5.2.1** and **5.2.2** shall be stated.

5.2.1 *Gauges* — A table of the pressure or the value of the ionic current obtained from the readings on the gauge to be calibrated, as a function of the pressure indicated by the reference gauge, shall be drawn showing in particular for each value of the electronic current and, if allowed by feed circuits in the assembly, for each set of values of the potentials applied to the electrodes.

5.2.2 *Gauge Tubes Only* — A table of the pressure (or corresponding physical quantities) read on the instruments connected to the gauge to be calibrated, as a function of the pressure indicated by the reference gauge, shall be drawn showing for each set of values of the variable parameters for which calibration is required.

5.3 From the tables, the linear range(s) for *n* percent* of the gauge is determined and the sensitivity giving the greatest accuracy within this range is derived from it.

5.4 In the case of complete gauge, for a calibration point (for example, the point corresponding to the sensitivity determined as indicated in **5.3**) the variations of this sensitivity for variations of the supply voltage of the electric mains within the range indicated by the manufacturer are determined.

APPENDIX A

(*Clauses* 2.1, 3.4 *and* 5.1)

CALIBRATION INSTALLATION

A-1. CALIBRATION INSTALLATION

A-1.1 The principle of the calibration installation is represented in Fig. 1.

A-1.2 Although the calibration bench will usually be made of metal, the conditions of **3.2.1** to **3.2.6.1** permit the use of a simple glass mounting which can be used for glass tabulated vacuum gauges.

A-1.3 In case the calibration gas is condensable with respect to the use of cold traps or a McLeod gauge as a reference, special precautions may be taken.

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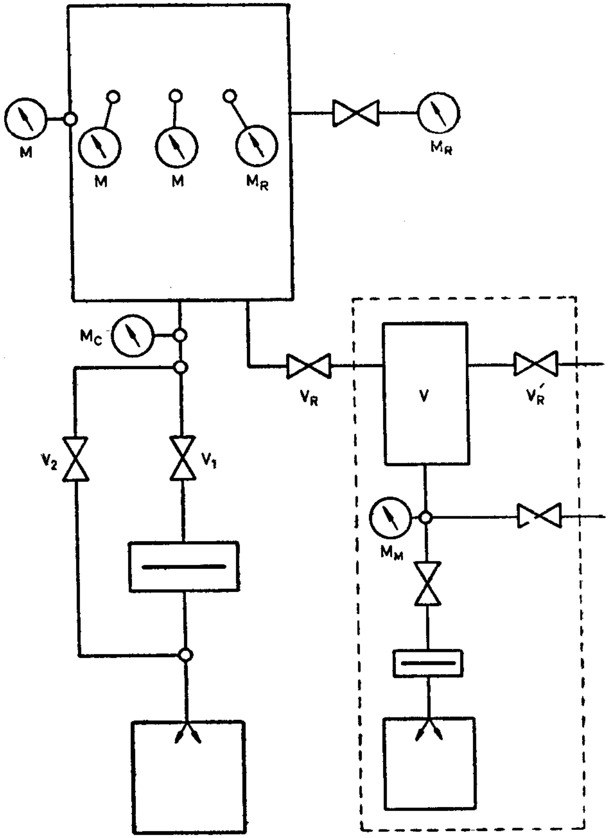
*A value to be determined.



M_R — Reference gauge — A valve is needed if the calibration gas is condensable and the gauge cannot be used under these conditions
M — Gauge to be calibrated
M_C — Ionization gauge, monitor for chamber pressure
M_M — Monitor gauge for pressure indication in buffer volume
V_1 — Pump adjustment valve
V_2 — By-pass to be used if calibration gas is condensable
V_R, V'_R — Gas inlet adjustment valves
V — Buffer volume

Optional — A stable and easy adjustment of the working pressure may be obtained by means of a buffer volume V, where the pressure is, for example, 100 to 1 000 times the calibration pressure. The pressure in V is measured with M_M. V_R may be replaced by a porous plug.

FIG. 1 CALIBRATION INSTALLATION

A-1.4 A transfer gauge is needed if the reference gauge cannot be used under these conditions. The readings of this transfer gauge shall be independent of the nature of the gas. The procedure to be carried out is as follows:

a) The transfer gauge shall be calibrated against the reference gauge with nitrogen gas, according to the procedure given in **4**. The valve to the cold trap and the reference gauge shall be closed.

b) The condensable vapour shall be admitted to the calibration chamber as required to establish the pressures desired for a test point.

c) The gauges shall be calibrated over the desired range of pressures following the procedures detailed in **4** using, however, the transfer gauge in place of the reference gauge.

(*Continued from page* 2)

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